Synthesis and Characterization of Novel Nanomaterials for Removal of Textile Dyes in Industrial Wastewater

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Declaration

I hereby declare that to the best of my knowledge this submission is my own work and it neither contains direct material previously published nor written by another person or material which to substantial extent has been accepted for the award of any other academic qualification of a university or other institute of higher learning except where acknowledgement is made in the text.

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ABSTRACT

Inefficient treatments and uncontrolled pollutants generation to the environment has been significantly affected the living standards in the ecosphere. The advancement in nanotechnology resulted in the effective treatment of all of the pollutants generated in the environment. In the field of textile dye removal, the special features of nanomaterials are gaining attention due to their enhanced physical, chemical and mechanical properties. Rice husk is an agricultural waste material that is used for the production of adsorbents in this study. Mesoporous silica nanoparticles were successfully synthesized by using rice husk as the raw material via a sol-gel pathway using cetyltrimethylammonium bromide (CTAB) as the structure-directing agent. The functionalization of silica nanoparticles was taken place in two pathways, such as in-situ and post functionalization methods by using 3-aminopropyltriethoxysilane (APTES) as the functionalization agent. Mesoporous silica nanoparticles were able to effectively adsorb methylene blue dye from aqueous solutions. The adsorption of MB could be best described by the pseudo-second-order model and well fitted to the Langmuir equation, with a maximum monolayer capacity of 19.26 mg/g. Photocatalytic decomposition of the organic pollutants gains emerging attention after the discovery of water splitting ability by TiO_2. The high purity (98.8%, TiO_2) rutile nanoparticles were successfully synthesized using ilmenite sand as the initial titanium source. This novel synthesis method was cost-effective and straightforward due to the absence of the traditional gravity, magnetic, electro statistic separation, ball milling and smelting processes. Also, highly corrosive environmentally hazardous acid leachate generated during the leaching process of ilmenite sand was effectively converted into highly efficient photocatalysts. The most efficient photocatalysts were composed of anatase-TiO_2/rutile-TiO_2/Fe_2O_3, α-Fe_2O_3/Fe_2TiO_5/TiO_2 and Fe_2TiO_5/TiO_2. The synthesized nanocomposites were characterized by microscopic (SEM and TEM) and spectroscopic (XRD, Raman, XPS, FT-IR and DRS) analytical techniques. These nano heterostructures were catalytically active for the photodegradation of methylene blue upon irradiation by a light source (LED or sunlight). Efficient charge separation and limiting electron-hole recombination in photocatalyst surfaces resulted in the overall performance of synthesized material. Reduced

graphene oxide (r-GO) has lately attracted a lot of attention to overcome limitations associated with photocatalysts. The honeycomb sp^2 network structure of r-GO improves charge separation and transportation through the surface of the catalyst. The fabricated GO/Fe_3O_4 heterogeneous photocatalyst shows very efficient degradation performance by overcoming the limitations associated with the narrow bandgap of Fe_3O_4 (0.1 eV). Finally, the synthesis of graphene like materials by catalytic graphitization of sucrose by using Fe and Ti transition metals studied in this study. Herein, we reported that Fe and Ti metal oxides promoted to the graphitization process at low temperature. Sucrose mixed with only uncalcined Fe_2O_3 produced Fe_3C, Fe, Fe_3O_4 dispersed on graphitic carbon, while sucrose mixed with only Fe_2TiO_5/TiO_2 product, and mixed with uncalcined Fe_2O_3 and Fe_2TiO_5/TiO_2 led to the production of TiO_2, Fe_3C, Fe, Fe_3O_4 dispersed on graphitic carbon. The most outstanding photocatalyst synthesized material composed with $TiO_2/Fe_3C/Fe/Fe_3O_4$–Graphitic carbon which is sun light sensing photocatalyst.

Keywords: Heterostructures, Rice husk, Reduced graphene oxide, Photocatalysis, Catalytic graphitization, Ilmenite sand

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Abbreviations

MSN	Mesoporous silica nanoparticles
CTAB	Cetyltrimethylammonium bromide
APTES	3-Aminopropyltriethoxysilane
RH	Rice husk
RHA	Rice husk ash
MSN-A	Mesoporous silica nanoparticles by sol-gel pathway
MSN-B	Amine modified MSN via co-synthesis method by pre addition of APTES
MSN-C	Amine modified MSN via co-synthesis method by post addition of APTES
MSN-D	Amine modified MSN via post grafting method
TiO_2-A	800 °C annealed amorphous TiO_2
TiO_2-B	800 °C annealed CTAB assigned amorphous TiO_2
TFTO	Uncalcined TiO_2/Fe_2TiO_5
TFTO-800	800 °C annealed TFTO powder TFTO
TF	Uncalcined TiO_2/Fe_3O_4
TF-450	450 °C annealed TF powder
TF-800	800 °C annealed TF powder
FFO-600	600 °C annealed Fe_2TiO_5/TiO_2
FOO-600	600 °C annealed α-Fe_2O_3
FOO-800	800 °C annealed α-Fe_2O_3
SF	Carbon and Iron composite

SI	Carbon and Ilmenite composite
SIF	Carbon, Ilmenite and Iron composite
LED	Light eliminative diode
r-GO	Reduced graphene oxide
GO	Graphene Oxide
SPR	Surface plasmon resonance
LSR	Localized surface plasmon resonance
DET	Direct electron transfer
FE-SEM	Field Emission Scanning Electron Microscope
FTIR	Fourier Transformation Infrared Spectroscopy
TGA	Thermogravimetric analysis
XRD	X-ray diffraction
XRF	X-ray fluorescence analyzer
XPS	X-ray photoelectron spectroscopy
TEM	Transmission electron microscopy
EDS	Energy dispersive spectra
DI	Deionized water
AHEAD	Accelerating Higher Education Expansion and Development
MOF	Metal-organic frameworks
EOM	Electronic, optical, and magnetic materials
SLINTEC	Sri Lanka Institute of Nanotechnology
ITI	Industrial Technology Institute